

VISIT...

LANZAROTE
Caliente.COM

RECRYSTALLIZATION: MICROSCALE

14

Microscale recrystallizations take place in the same way the big ones do. You just use teeny-tiny glassware. Read the general section on crystallization first. Then come back here and:

1. Put your solid in a small test tube.
2. Add a **microboiling stone** or a tiny **magnetic stirring vane** if you're on the stirring hot plate.
3. *Just cover* the solid with recrystallization solvent.
4. Heat this mix in the sand bath; stir with the spinbar or shake the test tube.
5. If the solid isn't dissolving, *consider*:
 - a. The stuff not dissolving is insoluble trash; adding lots more solvent won't *even* dissolve it.
 - b. The stuff not dissolving is your compound; you need to add 1-2 more drops of solvent.

This is a decision you'll have to make based on your own observations. Time to take a little responsibility for your actions. I wouldn't, however, add much more than 2–3 ml at the microscale level.

6. OK. Your product has *just dissolved* in the solvent. Add another 10% or so excess of solvent to keep the solid in solution during the hot filtration. If you're going to use *decolorizing carbon*, do it now. Add the carbon; the whole tube should turn black. Heat for 5–10 seconds, and then use Pasteur pipets to filter. You might have to filter twice if you've used carbon.
7. Get your solution into a test tube to cool and recrystallize. Let cool slowly to room temperature first; then put the tube in an ice-water bath for final recrystallization.
8. "I didn't get any crystals." Heat the tube and boil off some of the solvent. (**Caution—Bumping!**) Use a microboiling stone and don't point the tube at anyone. Repeat steps 7 and 8 until you get crystals. The point is to get to *saturation* while hot (all the solid that can be dissolved in the solvent is so), and the crystals will come out when cold.

If you've compared this outline to the large-scale recrystallization, you'll find that, with one exception—keeping a reservoir of hot solvent ready—the only difference here is the size of the equipment. Test tubes for flasks, filter pipets for funnels with paper, and so on. Because you can easily remove solvent at this scale, adding too much solvent is not quite the time-consuming boiling-off you'd have to do at the larger scale.

ISOLATING THE CRYSTALS

First, see the section on “Pipet Filtering—Solids” in Chapter 7 where you remove solvent with a pipet. I also mention using a Hirsch funnel. Just reread the section on the Buchner funnel filtration (Chapter 13, “Recrystallization”), and where you see “Buchner” substitute “Hirsch” and paste copies of the Hirsch funnel drawing over the Buchner funnel. Just realize that the tiny disk of paper can fly up more easily. Think small.

CRAIG TUBE FILTRATION

Usually this is called Craig tube crystallization, because you’ve pipet filtered your hot solution into the bottom of a Craig tube. So if you’ve recrystallized in something else, re-dissolve the crystals and get this solution into the bottom of a Craig tube (Fig. 68).

1. Carefully put the upper section of the Craig tube into the bottom section. Let the solution slowly cool to room temperature and then put the tube in an ice-water bath.

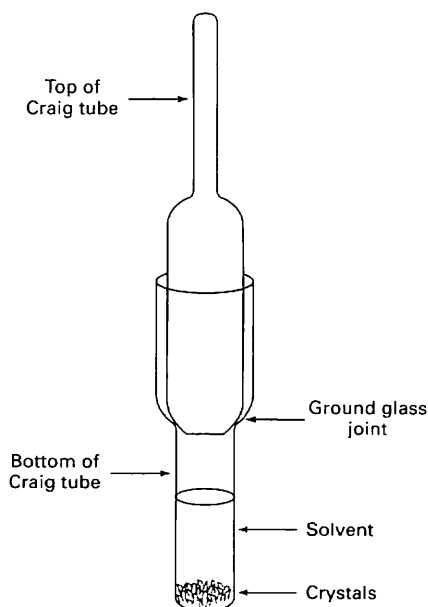


Fig. 68 Crystals in Craig tube.

2. Now the fun part. You've got to turn this thing upside down, put it in a centrifuge tube, and *not* have it come apart and spill everything. Easy to say—"Solvent is now removed by inverting the Craig tube assembly into a centrifuge tube..." (Mayo et al., p. 80). Easy to do ... well:
3. Get your centrifuge tube and cut a length of copper wire as long as the tube. Make a loop in the end that's big enough to slip over the end of the Craig tube stem. Twist the wire so that the loop won't open if you pull on it a bit.
4. Bend the loop so that it makes about a right angle from the wire (Fig. 69). Ready?
5. Hold the bottom of the Craig tube between your thumb and forefinger. Put the loop over the end of the tube.
6. Hold the wire gently between your thumb (or forefinger) and the glass tube bottom. Grip the hanging wire and gently pull it downward so that the Craig tube top is pulled into the bottom (Fig. 70). Too much force here and you could snap the stem. Watch it.
7. Once this is adjusted, tightly press the wire to the glass, so that the wire will keep the Craig tube closed when you turn the tube upside down.
8. Put the centrifuge tube upside down over the top of the Craig tube (Fig. 71b). Watch it. If the fit is snug, the copper wire could cause

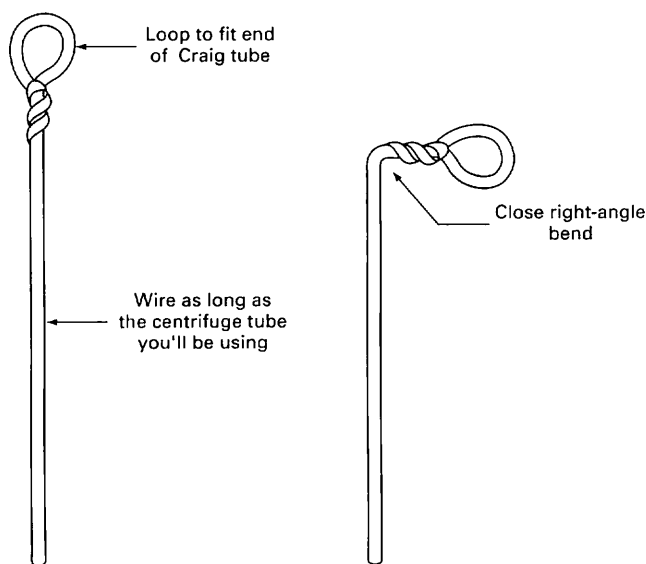


Fig. 69 Preparing wire loop for Craig tube.

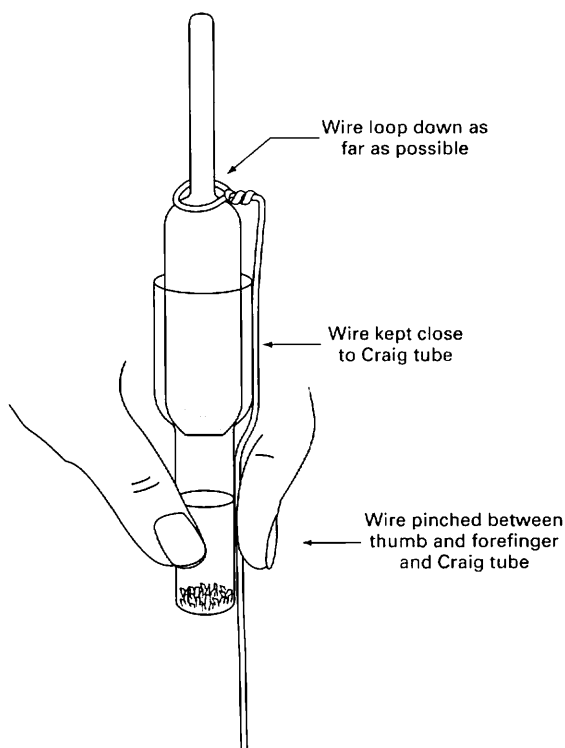


Fig. 70 Wiring the Craig tube.

problems, such as breaking the tubes. If the centrifuge tube is too tight, you might consider getting a wider one.

9. Once you get to this point, you may have three situations:

Baby Bear (Fig. 71a): The centrifuge tube is a bit shorter than the Craig tube. While not the best, some of the Craig tube peeks out from the centrifuge tube. The centrifuge *might* have clearance in the head to accommodate the extra length. I don't like this. Perhaps you should ask your instructor. Invert the setup and get ready to centrifuge.

Mama Bear (Fig. 71b): The centrifuge tube is a bit longer than the Craig tube. "A bit" here can vary. I mean you can push the Craig tube up into the centrifuge tube with your fingers so that the Craig tube stem touches the bottom or is within a centimeter or so of the bottom. Push the tube up as far as it will go. Invert the setup and get ready to centrifuge.

Papa Bear (Fig. 71c): The centrifuge tube is a *lot* longer than the Craig tube. You can't push it up to the bottom with your fingers. Grab the copper wire at the end, invert the setup, and quickly *lower* the Craig tube to the bottom of the centrifuge tube. Now you're ready to centrifuge.

CENTRIFUGING THE CRAIG TUBE

Just a few things here:

1. "Where does the centrifuge go when it takes a walk?" The centrifuge must be *balanced*. An even number of tubes, almost identical weights, set in positions opposite each other.
2. If you've cut the copper wire properly, it should stick out only a little bit from the centrifuge tube. You wouldn't want to be flailed by a foot-long

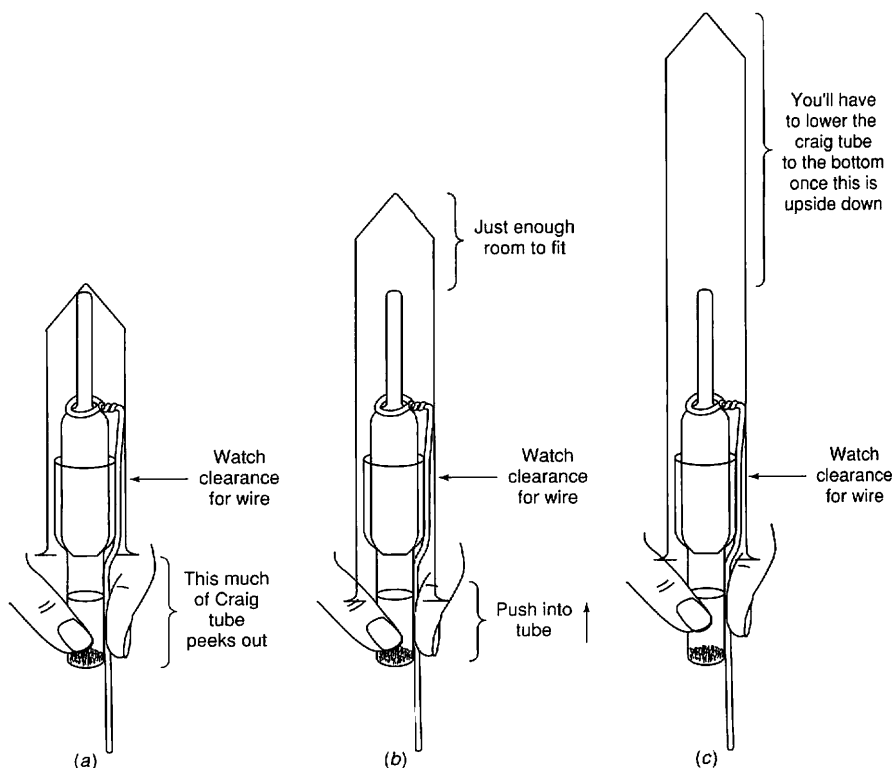


Fig. 71 The Three Bears of centrifuge tubes meet the Craig tube.

copper wire, would you? If necessary, bend it out of the way. Don't bend it so that you lose the wire into the centrifuge tube and can't get it again.

3. If centrifuging one minute is good, centrifuging 15 minutes is *NOT* better. Whatever the directions call for, just do it. Your yield will not improve. Trust me.

Getting the Crystals Out

After you've centrifuged the Craig tube, and the centrifuge has stopped turning

Do not stop the centrifuge with your fingers! Patience!

Pull the centrifuge tubes out, and carefully lift out the Craig tube. The solvent will have filled the bottom of the centrifuge tube, and the crystals will be packed down near the Craig tube top (Fig. 72).

I'd get a glass plate or watch glass, open the Craig tube, and put both sections on the glass to dry. (**CAUTION**—rolling Craig tubes will spread your product on the bench.)

After a bit, you can scrape your crystals into the appropriate tared container. (See "Tare to the Analytical Balance" in Chapter 6, "Microscale Jointware.") Don't try to get all of them; it's usually not worth the effort.

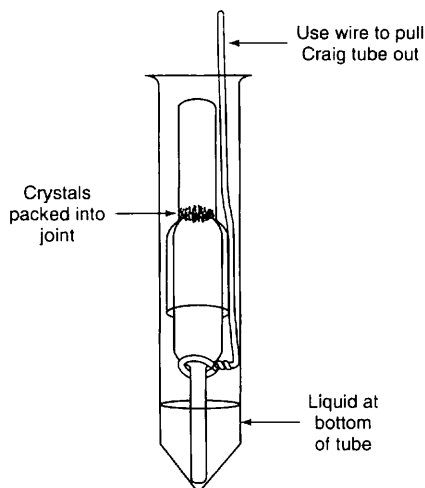


Fig. 72 Craig tube crystals after centrifugation.